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TI - Mold release slurry containing boron nitride and silica colloid for dental materials

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AB - The slurry, which is applied to the surface of molds to promote releasability of dental materials from the molds, comprises BN 5-60, silica colloid 1-20, resins sol or dispersible homogeneously in solvents 1-20, and org. solvents 50-90 wt.%. An acrylic stick was coated with a slurry prepd. from Me<sub>2</sub>CHOH 30, poly(vinyl butyral) 2000L 4, BN powder 20, Ca metaphosphate powder 10, OSCAP (colloidal silica powder) 6, and pine oil 30 parts and fixed on a rubber mount with wax. Subsequently a metal ring was placed around the stick and the inside space was filled with a slurry of a dental investment, followed by heated at 700.degree. to give a mold. A glass material was casted in the mold to give a semi-lucent and homogeneously vitrified glass stick with smooth surface and the shape, which was precisely reproduced.

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## PATENT ABSTRACTS OF JAPAN

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## (54) RELEASING MATERIAL SLURRY

## (57)Abstract:

PURPOSE: To produce a crystallized glass dental material good in dimensional accuracy by forming a uniform and stable reaction suppressing layer containing boron nitride on the surface of a molding mold material.

CONSTITUTION: This releasing material slurry is composed of 5-60wt.% boron nitride, 1-20wt.% silica colloid, 1-20wt.% resin soluble to a solvent or uniformly dispersible in the solvent, 50-90wt.% org. solvent consisting of a mixture of high volatile org. solvent and low volatile org. solvent and 2-30wt.% calcium phosphate crystal.

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CLAIMS

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[Claim(s)]

[Claim 1] 1 - 20 % of the weight of resin which can be distributed at homogeneity at soluble or a solvent to 5 - 60 % of the weight of boron nitride, 1 - 20 % of the weight of silica colloid, and a solvent, the release agent slurry which consists of 50 - 90 % of the weight of organic solvents.

[Claim 2] The release agent slurry of claim 1 whose organic solvent is the mixture of the organic solvent of high volatility, and the organic solvent of low volatility.

[Claim 3] The release agent slurry of claim 1 which contains the calcium phosphate crystal with which a calcium-metaphosphate crystal or strontium dissolved two to 30% of the weight, or claim 2.

[Claim 4] claims 1-3 used for production of the mold material used for manufacture of calcium phosphate system glass-ceramics dental materials -- the \*\*\*\*\* slurry of any 1.

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## DETAILED DESCRIPTION

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### [Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the release agent slurry of the mold used in case the dental materials by the release agent slurry, especially calcium phosphate system glass ceramics are fabricated.

[0002]

[Description of the Prior Art] In recent years, the dental materials of various ceramic systems are proposed and practical use is being presented with the part. Calcium phosphate system glass ceramics have the descriptions, like excelling in the moldability, and there is a close resemblance between physical properties and a natural tooth among these.

[0003] Since the viscosity of glass melt is low rich in a fluidity, while the calcium phosphate system glass-ceramics ingredient is easy to fabricate, in case it fabricates, it tends to react with mold material. Then, the method of preparing the reaction control layer which contains boron nitride 1% of the weight or more in the front face of mold material is proposed (refer to JP,1-10447,B).

[0004] Moreover, when heat-treat the fabricated calcium-phosphate system glass in mold material and carrying out crystallization processing, in that the glass ceramics which were excellent in a mechanical property or aesthetics when the crystal of a calcium metaphosphate was made to exist in the front face of mold material are obtained stably (refer to JP,2-56291,B), and the calcium-phosphate system crystallization glass of the presentation which contains a strontium oxide further, it is proposed that the mold material in which the calcium-phosphate crystal with which strontium dissolved was made to exist is effective (refer to JP,4-231344,A).

[0005]

[Problem(s) to be Solved by the Invention] When a suitable binding material and the suitable aggregate for boron nitride are added to the front face of die material as a simple approach for preparing the reaction control layer containing boron nitride, and this is applied to it on the surface of a mold or it is based on a lost wax process etc., after applying to the front face of the patterns (wax pattern etc.) of a mold face and a pattern is buried with a mold base material, there is a method of removing a pattern by the suitable approach. however, in order to use it for such a purpose, the homogeneity and stability obtain only by mixing an only required component – having – hard – the front face of a Plastic solid – a problem also tends to arise description – it came out. This invention aims at offering the release agent for preparing the reaction control layer containing boron nitride in the front face of die material etc.

[0006]

[Means for Solving the Problem] This invention offers the release agent slurry which becomes 5 - 60 % of the weight of boron nitride, 1 - 20 % of the weight of silica colloid, and a solvent from 1 - 20 % of the weight of resin which can be distributed to homogeneity at soluble or a solvent, and 50 - 90 % of the weight of organic solvents.

[0007] This release agent slurry forms a layer in the front face of mold material, and uses it for making good the mold-release characteristic of die material and fabricated material. A release agent slurry can form a mold release layer with thermal resistance by calcinating and removing an organic component. Although die material and especially fabricated material are not limited, glass can be used suitable for the mold material in the case of carrying out casting shaping, for example.

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[0008] Boron nitride is indispensable in order to give a \*\* form operation of fabricated material and mold material. Less than 5 % of the weight of the mold release effectiveness is [ the content of boron nitride ] insufficient, and it is unsuitable at it. A mold-release characteristic increases so that there are many contents of boron nitride, but since the fluidity of a release agent slurry will become inadequate and the mechanical strength after hardening will also fall if it exceeds 60 % of the weight, it is unsuitable. Specifically, the powder of the boron nitride of hexagonal system expressed with the empirical formula of BN is desirable as boron nitride.

[0009] Silica colloid needs to blend 1 % of the weight or more as solid content, in order to become the binder of boron nitride and to maintain reinforcement, when it heat-treats. However, since a mold release operation of boron nitride can weaken if there is too much silica colloid, the amount of silica colloid needs to be 20 or less % of the weight as solid content. The more desirable content of silica colloid is 3 - 10 % of the weight.

[0010] The thing in the condition that use the powder-like thing or the solvent distributed may be used for silica colloid. As a solvent, it is silica colloid with the desirable thing of an alcoholic system. In the case of powder, that whose diameter of a primary particle is about 1-100nm is desirable. In the case of dispersion liquid, that whose diameter of a particulate material of a silica is about 0.1-50 micrometers is desirable.

[0011] A resinous principle maintains the homogeneity of a slurry and has the work which raises the operability in the time of spreading etc. It is more desirable to avoid what generates gas harmful [ that what is necessary is just what is distributed to homogeneity ] to an organic solvent meltable as a resinous principle when heat-treating. For example, the ethyl cellulose which consists of various molecular weight, ethyl hide ROKISHI ethyl cellulose, a nitrocellulose, or a polyvinyl butyral can use preferably. Although the amount of a resinous principle is the balance of the class of resin and the class of solvent, and an amount and it is desirable to adjust so that it may have moderate viscosity, when fewer than 1 % of the weight, the improvement effectiveness in slurry homogeneity is not acquired. When exceeding 20 % of the weight, the release agent after heat-treatment becomes easy to collapse and is not desirable. The more desirable content of a resinous principle is 2 - 10 % of the weight. Two or more resin can also be mixed and used for the purpose, such as viscosity control.

[0012] Sufficient amount to dissolve or distribute resin to homogeneity, and distribute boron nitride, silica colloid, and other components is required for an organic solvent, and 50 % of the weight or more is required. Although the content of an organic solvent is adjusted according to the mold material which it is going to apply or the quality of the material of the pattern of a mold and a configuration, the method of application, etc., since the homogeneity of a slurry will be spoiled if it exceeds 90 % of the weight, it is unsuitable. It is desirable especially when the content of an organic solvent is 55 - 70 % of the weight.

[0013] Although it is low, it crawls on one kind again, and, as for an organic solvent, it is desirable shoes or to combine and use [ which are what has comparatively high volatility, and volatility ] it so that the suitable viscosity and the suitable rate of drying for a slurry may be given.

[0014] As an organic solvent of high volatility, the boiling point's thing 100 degrees C or less is [ the boiling point ] still more preferably desirable 150 degrees C or less. Specifically, with a carbon number [ of a methanol, ethanol, propanol, and a butanol ] of four or less alcohol is desirable.

[0015] As an organic solvent of low volatility, the boiling point's thing 200 degrees C or more is [ the boiling point ] still more preferably desirable 150 degrees C or more. Specifically, trimethyl pentanediol iso butyrate, diethylene glycol monobutyl ether acetate, and alpha-terpineol are mentioned. Besides a pure substance, the mixture of the natural product or composition like pineapple oil (oily liquid which uses as a principal component alpha-terpineol extracted from a pine), and a semisynthesis object can be used.

[0016] Adjusting suitably is desirable so that the rate of drying according to the purpose may be obtained, but in order to make stability distribute silica colloid, 10 - 60% of the weight of the whole slurry of the mixed rate of the organic solvent of high volatility and the organic solvent of low volatility is desirable also including parts, such as alcohol with which the amount of the organic solvent of high volatility is contained in a silica sol, and its 20 - 40 % of the weight is especially desirable. 5 - 50% of the weight of the whole slurry of the organic solvent of low volatility is

desirable, and its 10 - 40 % of the weight is especially desirable.

[0017] The release agent slurry of this invention can be used for shaping of various ingredients, such as a metal and glass. After carrying out casting shaping of the glass, it can also be made to crystallize as it is. When using the release agent slurry of this invention for the mold material of shaping crystallization of calcium phosphate system glass, it is desirable to make a calcium-metaphosphate crystal contain about 2 to 30% of the weight in a release agent slurry. The calcium-metaphosphate crystal blended into the release agent slurry acts as seed crystal in case a crystal deposits from glass, and equalizes the structure near the glass-ceramics front face, it is precise and glass ceramics with high reinforcement can obtain it. When there are few loadings of a calcium-metaphosphate crystal than 2 % of the weight, since there is a possibility that the effectiveness which makes crystallization homogeneity cannot fully demonstrate, it is not desirable. When the loadings of a calcium-metaphosphate crystal exceed 30 % of the weight, since there is a possibility of spoiling the mold release effectiveness of boron nitride, it is not desirable.

[0018] Moreover, unlike a calcium metaphosphate, the glass ceramics in which the crystal of a calcium metaphosphate with which strontium dissolved depending on the glass presentation deposited may be obtained, and, in such a case, it is desirable that the calcium-metaphosphate crystal as seed crystal also uses the calcium-metaphosphate crystal with which strontium dissolved similarly.

[0019] Furthermore, to a release agent slurry, it is the purpose which raises the dispersibility of boron nitride, and it is also possible to add surface activity material etc. suitably.

[0020] Although a release agent slurry is producible by mixing the above-mentioned combination component, it is required to fully mix each component. Although all components may be mixed at once, since especially resin and silica colloid have a possibility that it may not mix enough only by stirring, it is desirable to mix, after making an organic solvent dissolve or distribute each beforehand. As a mixed means, although based also on the viscosity of a slurry, the approach using the approach and ball mill using the approach, mortar, or mixer using a stirrer, and a roll mill etc. is employable. suitably. Moreover, mixing can also be promoted by heating suitably depending on the case. When there is a possibility that alcohol and other solvents may vaporize, it is good to use a container with a lid.

[0021] Various approaches, such as the approach of pulling up, after dipping the approach and mold which are sprayed on the front face of mold material by the approach of applying with the brush or a trowel, a spray, etc. as an approach of forming the layer which excelled [ front face / of mold material ] in the mold-release characteristic in this slurry using this release agent slurry, are employable. Moreover, it is also possible to produce all of mold material by this \*\*\*\*\* depending on the case. In order to raise the reinforcement on the front face of a mold as for which case, heat-treating at suitable temperature is desirable.

[0022] In the lost wax process well used for casting shaping of dental materials, the release agent slurry of this invention is applied to the pattern made from paraffin wax (wax pattern), and glass is cast to the opening which removed the wax pattern and was made into the part at the same time it is buried into the slurry of the die material called the investment, it heat-treats this and it stiffens mold material. Here, if the release agent slurry is applied to the front face of a wax pattern by various approaches before the above-mentioned flasking processing, when mold material will harden by heat-treatment, a release agent slurry is also calcinated and a mold release layer is formed in the front face of a mold base material.

[0023] Although it is desirable to adjust suitably according to the quality of the material of a Plastic solid-ed, magnitude, a configuration, molding temperature, etc. as for the thickness of a release agent layer, it is desirable that it is in the range of 10-300 micrometers.

[0024] A mold release layer needs to remove an organic component by heat-treatment in the case of shaping. When the organic component remains, air bubbles are involved in a glass Plastic solid by gaseous generating, or, in the case of the glass which is comparatively easy to be returned at an elevated temperature like calcium phosphate system glass, there is un-arranging [ of coloring by reduction occurring ]. The release agent layer after heat-treatment has the structure where seed crystal was combined with the silica binder, when boron nitride and seed crystal are used.

[0025]

[Example]

Polyvinyl butyral resin (DENKI KAGAKU KOGYO K.K. make and polyvinyl-butylal 2000L) was 4-weight-\*\*\*\*\* (ed) in the example 1 isopropanol 30 weight section, and was agitated in it, and it dissolved in it. This was put into the mortar, and the boron nitride powder 20 weight section, the calcium-metaphosphate crystal powder 10 weight section, and the colloid silica powder (catalyst formation industrial incorporated company make, trade name (OSCAP)) 6 weight section were added and mixed, and the pineapple oil 30 weight section could be added further, it mixed, and the release agent slurry was produced.

[0026] When the above-mentioned release agent slurry was applied to the rod with die length of 20mm, a width of face [ of 2mm ], and a thickness of 2mm made of acrylic resin, the slurry was smooth, and its mileage was good and was able to apply it to homogeneity thinly. This was stood on the base made of rubber, and it fixed with the soft wax (\*\*\*\* dentistry industrial incorporated company make, trade name utility wax). The metal ring was placed so that the acrylic resin rod which applied the slurry on the base made of rubber might be surrounded, after slushing and stiffening the slurry which melted dental investing material (Tokuyama Make, trade name blue best) powder with water in the ring, the rubber base was removed, it put into the electric furnace, and mold was produced by heat-treating at 700 degrees C.

[0027] P2 O5 which held this mold at 650 degrees C, and was fused at 1100 degrees C 67 % of the weight and CaO are 26 % of the weight and aluminum 2O3. 4 % of the weight and Ce 2O3 Centrifugal pressure casting cast 3% of the weight of glass, and as it is, with the mold, at 700 degrees C, it maintained for 16 hours and crystallized. It cooled to the room temperature after crystallization, and glass ceramics were taken out from mold material. The acquired glass-ceramics casting object was translucent, and it was crystallizing to homogeneity, and the front face was smooth and the configuration of a rod was reproduced correctly.

[0028] After having put the slurry into the container, sealing it for the preservation stability test of a slurry and saving for four months in a 40-degree C thermostat, mold was produced like the start and casting crystallization of the glass was carried out. Although the little supernatant part had arisen in the slurry, by stirring lightly, it could equalize easily and was able to apply smoothly thinly like the start. Moreover, the glass-ceramics casting object which has a beautiful appearance like the start was acquired.

[0029] two kinds of ethyl cellulose (the U.S. Hercules make, a trade name N-7, and N-22) from which an example dyad amount differs – respectively – as 1 % of the weight, 4 % of the weight and 10 % of the weight (DENKI KAGAKU KOGYO K.K. make and trade name polyvinyl-butylal 2000L) of polyvinyl butyral resin, and a solvent In the liquid 36 weight section which consists of 55 % of the weight of alpha-TEREPINE oar, and 30 % of the weight of diethylene glycol monobutyl ether acetate the silica binder liquid (a catalyst – formation – industrial incorporated company make –) which uses isopropanol as a solvent including about 30% of the weight of silica colloid the trade name OSCAL1432 18 weight section, the boron nitride 19 weight section, and P2 O5 \*\*\*\*\* – the calcium-metaphosphate crystal powder 9 weight section in which 50-mol % of a phosphoric acid, and the strontium which contains 13-mol % of strontium as 37-mol % of calcium SrO as CaO dissolved – The isopropanol 18 weight section was added, it fully mixed, and the release agent slurry was produced.

[0030] the slurry of the above [ pattern / of a crown configuration / wax ] – \*\*\*\* – the dental investing material (SHOFU Make and a trade name – uni--- the best – non – precious) after applying thinly and drying – using – a connoisseur – flasking and wax incineration were performed according to the method, and mold was produced.

[0031] P2 O5 which held this mold material at 650 degrees C, and was fused at 1100 degrees C 67% of the weight, CaO is 16 % of the weight, and SrO is 10 % of the weight and aluminum 2O3. 4 % of the weight and Ce 2O3 Centrifugal pressure casting cast 3% of the weight of glass, and as it is, with the mold, at 700 degrees C, it maintained for 16 hours and crystallized. It cooled to the room temperature after crystallization, and glass ceramics were taken out from mold material. The acquired glass-ceramics casting object was translucent, and it was crystallizing to homogeneity, and the front face was smooth and the configuration of a wax pattern was reproduced correctly.

[0032] After having put the slurry into the container, sealing it for the preservation stability test of a

slurry and saving for four months in a 40-degree C thermostat, mold was produced like the start and casting crystallization of the glass was carried out. Although the little supernatant part had arisen in the slurry, by stirring lightly, it could equalize easily and was able to apply smoothly thinly like the start. Moreover, the glass-ceramics casting object which has a beautiful appearance like the start was acquired.

[0033] It replaced with the calcium metaphosphate of the release agent slurry of example 3 example 2, the boron nitride powder of the same weight was added, and the release agent slurry was produced like the example 2 except having made boron nitride into a total of 28 weight sections.

[0034] P2 O5 fused at 1100 degrees C to the place which applied the above-mentioned release agent slurry inside the mold, and was calcinated at 700 degrees C for 1 hour 67% of the weight, CaO is 16 % of the weight, and SrO is 10 % of the weight and aluminum 2O3. 4 % of the weight and Ce 2O3 3% of the weight of glass was poured, and the block of glass was produced. After cooling, glass could be easily picked out from the mold and the glass block [ go out ] was obtained.

[0035]

[Effect of the Invention] The release agent slurry of this invention is smooth, and since spreading nature is good, it can form the mold release layer of thin uniform thickness in a mold front face. Moreover, it is possible for the rate of desiccation to control and the trouble produced at the time of desiccation of generating of a crack etc. can also be solved. When manufacturing calcium phosphate system glass ceramics using the mold which has the mold release layer formed by this release agent slurry, dimensional accuracy is high and dental materials are obtained for glass ceramics with a smooth front face.

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